

# PATENT ABSTRACTS OF JAPAN

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## (54) PRODUCTION OF DEXTRIN

### (57)Abstract:

**PROBLEM TO BE SOLVED:** To provide a method for producing a dextrin with easiness for performance of filtration and deionization, in high yield and with economical advantages, as compared with conventional production methods.

**SOLUTION:** This method comprises addition of water to corn starch to make its slurry having a solid concentration of 5-30wt.%, adjustment, with addition of calcium hydroxide, of the slurry to pH 9.5-12.4 under homogenous mixing, heating at 95-150°C followed by neutralization, and finally, enzymatic liquefaction to obtain a low-viscosity dextrin liquid.

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**CLAIMS**

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[Claim(s)]

[Claim 1] The manufacture approach of the dextrin which it faces manufacturing a dextrin, water is added to amylum maydis, and it considers as the slurry of 5 - 30 % of the weight of solid content concentration, adds a calcium hydroxide, adjusts to pH 9.5-12.4, heats at the temperature of 95-150 degrees C, mixing to homogeneity, and is characterized by going via an enzyme liquefaction process after neutralizing.

[Claim 2] The manufacture approach of a dextrin according to claim 1 of heating for 5 - 60 minutes and carrying out an enzyme liquefaction process to DE (dextrose equivalent) 1.5-15 at the temperature of 90-105 degrees C using the heat-resistant hankyu liquifase of one to 20 international unit (IU) per 1g of substrate solid content.

[Claim 3] Face manufacturing a dextrin, add water to amylum maydis, and it considers as the slurry of 5 - 25 % of the weight of solid content concentration. Mixing to homogeneity, add a calcium hydroxide, adjust to pH 10-12, and it heats for 5 - 60 minutes at the temperature of 105-135 degrees C. The manufacture approach of the dextrin characterized by going via a decolorization process, a deionization process, and a concentration process after adding heat-resistant hankyu liquifase, liquefying to DE 2-10 at the temperature of 90-98 degrees C after neutralizing, and carrying out deactivation of the enzyme by four or less pH.

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**DETAILED DESCRIPTION**

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[Detailed Description of the Invention]

[0001]

[Field of the Invention]

[0002] This invention relates to the approach of manufacturing a dextrin from starch.

[0003]

[Description of the Prior Art]

[0004] Generally, a dextrin is the generic name of the mixture of the decomposition product of the various polymerization degree acquired by hydrolyzing starch by the acid, an amylase, etc., many do not have a special structural feature and its molecular weight is not fixed, either.

[0005] Generally, it is classified into amylopectin (blue), erythropectin (red), achropectin (coloration is not carried out), a maltopectin (coloration is not carried out), etc. with extent of the coloration in an iodine starch reaction.

[0006] In recent years, paying attention to the endowment of mouthfeel snappily carried out at the time of using for low sweet taste, water retention, moderate viscosity, elasticity endowment for food, deep-fried dishes, etc., utilization is increasing also in the property of these dextrins.

[0007] As [ introduce / to \*\* JP,48-67447,A / as the manufacture approach of the conventional dextrin ] Starch is made into about 10 - 30 % of the weight of solid content concentration, and it is DE (the rate of the reducing power of the sample when setting reducing power of a dextrose equivalent and grape sugar to 100 is expressed.) with an acid or an enzyme. It hydrolyzes to 5-15 and liquefaction liquid is prepared. This Decolorization with activated carbon, How to dry after passing through the deionization in filtration and ion exchange resin, to heat-treat at further 170-300 degrees C for 5 minutes to 3 hours, and to obtain a dextrin, \*\* Make liquid generate. the saccharification which the beta-amylase is made to act on starch which is introduced to JP,52-46290,B, and mainly consists of a maltose and beta-limit dextrin -- the saccharification -- the approach of carrying out chromatography separation of the liquid with OH mold anion exchange resin, and manufacturing the maltose and beta-limit dextrin of a high grade -- \*\* Make liquid generate. the saccharification which alpha-amylase is made to act on it until it becomes starch which is introduced to JP,61-205494,A at about 25 DE, and consists of branching dextrins and straight chain oligosaccharide -- subsequently, the saccharification acquired -- there was the approach of carrying out selection judgment of branching dextrins and the straight chain oligosaccharide etc. by contacting liquid on the ion-exchange resin of a gel mold.

[0008] However, many technical problems were left behind to the manufacture approach of the conventional dextrin. For example, although the filtration process was required after the decolorization in a production process, when it was going to obtain the product low [ DE ] which the latest user desires, viscosity became high extremely, and filtration by economical concentration was difficult for the approach of the aforementioned \*\*. Moreover, since the product which the phenomenon which forms a coat on the surface of ion exchange resin in the case of deionization occurs, and the technical problem that ion exchange resin loses a deionization function in the inside of a short time extremely also has, and was obtained by this approach presented deep yellow thru/or brown when it dissolved in water, it also had the technical problem that an application was limited.

[0009] Moreover, the dextrin component which the technical problem that concentration costs increase occurs in the case of subsequent commercial production since operation by very low concentration is required of the approach of the aforementioned \*\* or \*\* in case chromatography separation of the component is carried out with ion-exchange resin at a dextrin fraction and an oligosaccharide fraction, and cannot be separated since the

sharpness of separation is not high in case it is chromatography separation entered into the oligosaccharide fraction, and the technical problem that the recovery of a dextrin was low also had it.

[0010]

[Means for Solving the Problem]

[0011] The result to which this invention person etc. studied the manufacture approach of a dextrin wholeheartedly in order to solve said technical problem, After adjusting, heating and neutralizing to pH 9.5-12.4, by adding a calcium hydroxide to the slurry of amylum maydis, and going via an enzyme liquefaction process by the remarkable short simple approach It succeeds in preparing the dextrin equipped with the property suitable for a food-stuff-industry application which was excellent in versatility, and came to complete this invention.

[0012] The first this invention is faced manufacturing a dextrin, adds water to amylum maydis, makes it the slurry of 5 - 30 % of the weight of solid content concentration, adds a calcium hydroxide, adjusts it to pH 9.5-12.4, mixing to homogeneity, is heated at the temperature of 95-150 degrees C, and after neutralizing, it is the manufacture approach of the dextrin characterized by going via an enzyme liquefaction process.

[0013] The second this invention is the manufacture approach of a dextrin given in said first invention which heats for 5 - 60 minutes and carries out an enzyme liquefaction process to DE (dextrose equivalent) 1.5-15 by within the limits with a temperature of 90-105 degrees C using the heat-resistant hankyu liquifase of one to 20 unit per 1g of substrate solid content.

[0014] Face the third this invention manufacturing a dextrin, it adds water to amylum maydis, and makes it the slurry of 5 - 25 % of the weight of solid content concentration. Mixing to homogeneity, add a calcium hydroxide and it adjusts to pH 10-12. After adding heat-resistant hankyu liquifase, liquefying to DE 2-10 at the temperature of 90-98 degrees C, after heating for 5 - 60 minutes and neutralizing at the temperature of 105-135 degrees C, and carrying out deactivation of the enzyme by four or less pH, it is the manufacture approach of the dextrin characterized by going via a decolorization process, a deionization process, and a concentration process.

[0015]

[Embodiment of the Invention]

[0016] The content of this invention is explained below at a detail.

[0017] The quality of the amylum maydis of extent which can adopt it advantageously if the starch used for this invention is starch of the corn origin, and constraint does not have in the manufacture approach of the place of production of raw material corn or starch, is marketed as corn starch, and is used as a common raw material for starch sugar manufacture is enough.

[0018] Since a dextrin and other components may not dissociate clearly in liquid but separation may become difficult in the case of a filtration process after adding the calcium hydroxide in this invention and heat-treating pH by 9.5-12.4 when this invention is applied to starch of the tapioca origin other than corn (for example, a potato), the activity of the starch of these origins should be avoided.

[0019] Although it considers as the slurry-like mixture which adds water to amylum maydis and is called starch milk in case this invention is carried out, the solid content content in that case has 5 - 30 desirable % of the weight, and 5 - 25% of the weight of its range is still more desirable.

[0020] Although it is possible to perform the reaction of this invention when solid content concentration is less than 5% Are not desirable from the economical reasons of concentration costs increasing at that the throughput per magnitude of a facility becomes small, or a next process. When exceeding 30%, it is not desirable too from the reasons of actuation of heating, churning when viscosity becomes very high during churning, unevenness is made to a reaction or this invention is carried out for it, migration, filtration, etc. becoming very difficult.

[0021] Although the calcium hydroxide used for this invention is marketed as a food additive, if it has quality, it is enough, and there is no constraint in the gestalt and it can adopt advantageously all of the shape of the shape of a liquid and a slurry, and powder as it.

[0022] Next, the approach using a jet cooker which mixes the live steam and this slurry which are generally used as an approach of mixing to homogeneity this slurry obtained above in the case of starch liquefaction in an instant, and is made to pile up within a reaction etc. can adopt advantageously, and any of a batch process and continuous system are sufficient also as the method.

[0023] Although one of the big descriptions of this invention is to adjust within the limits of 10-12 still more

preferably, and heat [ 9.5-12.4, and ] within the limits of 105-130 degrees C still more preferably the temperature of 95-150 degrees C, pH of this starch slurry Since the effectiveness of this invention which is mentioned later, without a reaction fully advancing may not fully be acquired and a calcium hydroxide is preferably used by this invention on the other hand when pH in this case is less than 9.5 When pH exceeds 12.4, it is not desirable from the reasons of other pH regulators' being required and side reaction occurring.

[0024] Moreover, although whenever [ stoving temperature ] has desirable 95-150 degrees C when acquiring the good effectiveness of this invention, and 105-130 degrees C is still more desirable, when a reaction does not fully advance in the case of less than 95 degrees C but it exceeds 150 degrees C, bad debt and side reaction may occur.

[0025] Although the acid used in case a starch sugar ghost is generally prepared can adopt advantageously the neutralizer in the case of neutralizing after heating in this invention, since it is advantageous to carry out precipitate clearance of the calcium used previously when the process after neutralization is taken into consideration, an acid which combines with calcium and generates precipitate, for example, oxalic acid, a sulfuric acid, phosphoric acid, etc. are the most advantageous.

[0026] although it is desirable to make it advantageous pH range in the case of next enzyme liquefaction as a rule of thumb of pH when neutralizing -- the range 6.0-6.9 where alpha-amylase is stable as desirable pH and where activity is high, i.e., pH, -- the range of pH 6.5-6.8 is raised still more preferably.

[0027] Next, although the various alpha-amylases adopted in the case of a general starch liquefaction process can adopt advantageously the hankyu liquifase used in this case although it is making to go via an enzyme liquefaction process into indispensable requirements in this invention, especially the heat-resistant hankyu liquifase that can be used at temperature high also in them is advantageous, and Termamyl (trademark) for example, by the Novo industry company etc. is mentioned as a class of brand.

[0028] Although the additions of an enzyme are employable as arbitration if they are the need and sufficient amount when realizing this invention, from the range of an experience of an artificer, one to 20 international-unit (IU) extent is about suitable per 1g of starch solid content.

[0029] although the range where hankyu liquifase can demonstrate enzyme activity to advantageous extent economically should be adopted, in case heat-resistant hankyu liquifase is used for the temperature of an enzyme reaction still more preferably, it is still more suitable 80-108 degrees C as a desirable temperature requirement -- 90-105-degree C 90-98 degrees C are mentioned most preferably.

[0030] when [ moreover, ] acquiring the effectiveness of this invention -- an enzyme liquefaction reaction -- DE 1.5-15, although carried out to the range of 2-10 still more preferably In the case of less than 1.5 DE, it is very difficult to make desired DE suspend a reaction itself. and since the viscosity of the enzyme liquefaction liquid obtained is high, when it is common for handling to be difficult and it exceeds DE15 Since the property which is not desired as dextrans, like that the amounts of generation, such as a maltose and a glucose, increase and sweet taste becomes strong and viscosity becomes low too much becomes strong, it is not desirable.

[0031] Although the actuation to which deactivation of the enzyme is carried out is required after the enzyme liquefaction reaction of this invention Although each of approaches of the approach generally used in the starch sugar-ized industry being able to adopt the approach advantageously, for example, adding acids, such as a hydrochloric acid, a sulfuric acid, phosphoric acid, and oxalic acid, and lowering pH, approaches of heating and carrying out deactivation to about 110 degrees C, etc. can adopt When obtaining the product of fixed quality also in these, the method of adding an acid is the most desirable.

[0032] although it goes via a decolorization process in the desirable embodiment of this invention -- general saccharification of grape sugar, a starch syrup, etc. -- it is most desirable to be able to apply advantageously, also in case the approach adopted as elegance is this invention, and to decolorize by the approach of a batch process or continuous system using granular active carbon or powder-like activated carbon.

[0033] Although it is necessary to insert a filtration process after decolorization to adopt powder-like activated carbon, in the case of this invention, compared with the manufacture approach of the conventional dextrin, operation of a filtration process is very easy, and this is remarkable effectiveness acquired by carrying out this invention.

[0034] moreover, the usual saccharification in case a filtration process is carried out -- suitable filter aid, such as diatomaceous earth used at the time of manufacture of elegance, may be used, precoat can be carried out to filter-medium front faces, such as a filter cloth, it can mix on them with activated carbon, or performing above

both can also be advantageously adopted as them.

[0035] furthermore -- although it goes via a deionization process in the desirable embodiment of this invention - - saccharification general also in this case -- approaches, such as the ion-exchange material and batch processes which are adopted as manufacture of elegance, such as a zeolite and resin for deionization, and continuous system, can adopt in favor also of this invention.

[0036] if an example of the combination of the resin adopted in the case of a deionization process is given -- the order of the monobed of strongly acidic cation exchange resin, strongly basic anion exchange resin, weakly acidic cation exchange resin, the Nakashio machine nature anion exchange resin, strongly acidic cation exchange resin, and strongly basic anion exchange resin -- ion exchange resin -- it is also an advantageous deionization method to let a column pass.

[0037] Since the mucus film is not formed in the front face of that the viscosity of liquid is remarkably low, or deionization material in the case of this invention and the life of deionization material is not shortened compared with the manufacture approach of the conventional dextrin in the case of this deionization process, operation is very easy and it is the remarkable effectiveness acquired when this also carries out this invention.

[0038] Although the dextrin water solution refined at the aforementioned decolorization and a deionization process goes via a concentration process in the desirable embodiment of this invention If it is a suitable approach in case matter with comparatively high viscosity, such as a thin film flowing-down type which is generally adopted as manufacture of a starch sugar-ized product about the approach, for example, is condensed, are employable. Although what is necessary is just to condense to extent which the suitable concentration in the case of making it circulate as a liquefied product changed with DE of a product, and met want of a consumer, it is convenient when it is going to obtain a powder article, and it is made about 30 - 65% of concentration.

[0039] Moreover, after condensing, it is also free to prepare powder or granulation by the well-known approach in itself [, such as a spray drying method ] if needed.

[0040] By carrying out this invention explained above, rather than the conventional approach, it becomes possible to perform processes, such as filtration and deionization, very easily, and the dextrin which has the outstanding property can be obtained.

[0041]

[Example]

[0042] Although an example is hung up over below and the content of this invention is explained to it still more concretely, the technical range of this invention is not restricted by the following examples.

[0043] In addition, among each example, especially % shall express weight % altogether, unless it refuses.

[0044]

[Example -1]

[0045] Starch [Japan Maize Products Co., Ltd. make and corn-starch] 150g of marketing was put in into the proof-pressure liquefaction testing device with an agitator with a volume of 10l., and the calcium hydroxide of a reagent was added, it was referred to as pH10.5, agitating, after adding 850g of water and mixing, live steam was blown, and it heated for 15 minutes at the temperature of 125 degrees C.

[0046] Next, releasing and agitating the lid of a proof-pressure container, add oxalic acid and it is referred to as pH6.5. Adjusting in temperature of 95 degrees C Commercial heat-resistant hankyu liquifase [Novo industry company make, Add Termamyl (trademark)]750IU and it liquefies to DE5. Oxalic acid is added, deactivation of the enzyme was carried out, 1g of commercial powdered activated carbon was added, churning decolorization was carried out for 20 minutes at the temperature of 50 degrees C, diatomaceous earth filter aid [Showa chemistry incorporated company make and trade name radio light (trademark)] 1g of marketing was further added and filtered until it was set to pH3.9, and the filtrate was obtained.

[0047] Furthermore, deionization of this filtrate was carried out with ion exchange resin, it condensed to 30% of concentration with rotating type thin film type concentration equipment (the Tokyo Rikakikai Co., Ltd. make, a rotary evaporator, N-1 N type), and 480g of clear dextrin water solutions was obtained by colorlessness. (The yield of solid content is 96% when the starch of a raw material is set to 100.)

[0048] The property of the dextrin water solution obtained in the example -1 is explained below.

[0049] As a result of measuring at [viscosity (concentration)] temperature of 40 degrees C, the viscosity of the dextrin water solution obtained in the example -1 was 40cp by 6cp and 30% at 4cp and 20% in 10% of concentration at 12cp and 40%.

[0050] It was light orange as a result of presenting an iodine starch reaction with the dextrin water solution obtained in the [iodine reaction] example -1. This result shows that most components of large molecular weight do not remain by actuation of this invention.

[0051] As a result of storing the dextrin water solution of 30% of concentration obtained in the [trial of aging] example -1 at the temperature of 4 degrees C and measuring the permeability of the light of this water solution, it was 99.2%, and as a result of measuring till the 30th every 5th day, light transmittance did not fall but was 99.2%. This result shows having the outstanding property in which the dextrin obtained by this invention cannot age very easily during a retention period.

[0052] [Sugar composition (G shows a grape-sugar unit)] For G2, G3 was [ G1 / four or more / 8.1% G ] 86.9% 4.5% 0.5%.

[0053]

[Example -2]

[0054] Set the amount of 70g and water to 930g for the amount of starch, set pH after adding a calcium hydroxide to 10.9, and it considered as for 20 minutes at the temperature of 110 degrees C, and also heated like the example -1.

[0055] Next, neutralized like the example -1, set the amount of enzymes to 10IU per 1g of starch, and it considered as the temperature of 94 degrees C, and also enzyme liquefaction was carried out to DE=3 like the example -1, enzyme deactivation, decolorization, deionization, and concentration were performed, and the dextrin water solution was obtained.

[0056] In actuation, the filtration after decolorization is easy, actuation is easy also in the case of deionization, and there was no difficulty on actuation in any way.

[0057]

[Example -3]

[0058] Set the amount of 200g and water to 800g for the amount of starch, set pH after adding a calcium hydroxide to 10.8, and it considered as for 20 minutes at the temperature of 130 degrees C, and also heated like the example -1.

[0059] Next, neutralized like the example -1, set the amount of enzymes to 15IU per 1g of starch, and it considered as the temperature of 95 degrees C, and also enzyme liquefaction was carried out to DE=11 like the example -1, enzyme deactivation, decolorization, deionization, and concentration were performed, and the dextrin water solution was obtained.

[0060] saccharification usual in a filtration process in process and a deionization process -- it is possible to operate it like processing of elegance, and there was no difficulty on actuation by the high viscosity and the high mucilage like [ at the time of the usual dextrin manufacture ].

[0061] The property of the dextrin water solution obtained in the example -3 is explained below.

[0062] As a result of measuring at [viscosity (concentration)] temperature of 40 degrees C, the viscosity of the dextrin water solution obtained in the example -3 was 30cp by 5cp and 30% at 3.5cp and 20% in 10% of concentration at 10cp and 40%.

[0063] It was light orange as a result of presenting an iodine starch reaction with the dextrin water solution obtained in the [iodine reaction] example -3. This result shows that most components of large molecular weight do not remain by actuation of this invention.

[0064] As a result of storing the dextrin water solution of 30% of concentration obtained in the [trial of aging] example -3 at the temperature of 4 degrees C and measuring the permeability of the light of this water solution, it was 99.1%, and as a result of measuring till the 30th every 5th day, light transmittance did not fall but was 99.0%. This result shows having the outstanding property in which the dextrin obtained by this invention cannot age very easily during a retention period.

[0065] [Sugar composition (G shows a grape-sugar unit)] For G2, G3 was [ G1 / four or more / 10.3% G ] 79.9% 8.3% 1.5%.

[0066]

[Example -4]

[0067] Set pH after adding a calcium hydroxide to 10.3, and it considered as for 20 minutes at the temperature of 130 degrees C, and also [ all ] the dextrin water solution was obtained like the example -1.

[0068] saccharification usual in a filtration process in process and a deionization process -- it is possible to

operate it like processing of elegance, and there was no difficulty on actuation by the high viscosity and the high mucilage like [ at the time of the usual dextrin manufacture ].

[0069] The obtained dextrin water solution was further condensed under reduced pressure, and as a result of drying, the white powder-like dextrin was obtained.

[0070] the time of the obtained powder having not almost had sweet taste and there having been no starch smell, and it being easily dissolvable in chilled water, and dissolving in it -- colorlessness -- it became a clear water solution.

[0071]

[The example -1 of a comparison]

[0072] Heat-treatment when the amount of 320g and water was set to 680g and also a calcium hydroxide is added and heated like an example -1, while it cannot mix since viscosity is very high and agitates was not able to do the amount of starch.

[0073]

[The example -2 of a comparison]

[0074] After having set pH after adding a calcium hydroxide to 8.5, and also heat-treating like the example -1 and neutralizing, when enzyme liquefaction was carried out like the example -1, deactivation of the enzyme was carried out and the decolorization process was performed, the filter mucilage adhered and it was not able to filter.

[0075]

[The example -3 of a comparison]

[0076] After having made whenever [ stoving temperature / after adding a calcium hydroxide ] into 90 degrees C, and also heat-treating like the example -1 and neutralizing, when enzyme liquefaction was carried out like the example -1, deactivation of the enzyme was carried out and the decolorization process was performed, the filter mucilage adhered and it was not able to filter.

[0077]

[The example -4 of a comparison]

[0078] Mix commercial starch [Japan Maize Products Co., Ltd. make and corn-starch] 150g and 850 commercial of water, and it considers as 15% of solid content concentration. Per 1g of substrates, after adjusting to pH6.2 The heat-resistant hankyu liquifase [Novo industry company make of 10IU, After having heated for about 3 minutes at the temperature of 105 degrees C, then having cooled liquid at 65 degrees C using Termamyl (trademark)], adding this heat-resistant hankyu liquifase 10IU further and holding for 30 minutes, oxalic acid was added, it was referred to as pH3.8, and the dextrin of DE15 was obtained.

[0079] As a result of storing the dextrin water solution of 30% of concentration obtained in the example -4 of a comparison at the temperature of 4 degrees C and measuring the permeability of the light of this water solution, although it was immediately after manufacture about 98.4%, white muddiness already occurred at the 1st day, as a result of measuring till the 30th every 5th day, with time amount, light transmittance continued falling and permeability became about 15% on the 25th.

[0080]

[Effect of the Invention]

[0081] Since it remains into liquid with the gestalt which the enzyme liquefaction liquid obtained by carrying out this invention is hypoviscosity, and components other than a dextrin tend [ very ] to separate Compared with the manufacture approach of the conventional dextrin, implementation of manufacture actuation, such as filtration and deionization, becomes very easy. moreover -- since the product recovery per raw material is alike and high compared with the approach of carrying out fractionation and obtaining a dextrin and it is economically advantageous, after carrying out an enzyme reaction superfluously -- easy \*\*\*\* of operation -- the manufacture approach of an advantageous dextrin is offered economically.

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[Translation done.]